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SUMMARY OF PLUTONIUM OXIDE AND METAL STORAGE PACKAGE FAILURES

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Abstract
This article compiles available documented information on failures of containers used to store plutonium-bearing oxide and metal materials within the context of the U. S. Department of Energy (DOE) stabilization, packaging and storage standard DOE-STD-3013. Relevant information was obtained from published DOE-wide plutonium storage safety evaluations, workshops, technical reports, scientific journal publications and direct discussion with many subject matter experts. This article focuses on the past two to three decades of plutonium oxide and metal storage, during which package failures were reasonably well documented. Storage of residues and wastes is not covered in this study.

Based on the documented information examined, this report identifies two dominant failure modes for plutonium oxide and metal storage package failure:

- Metal oxidation due to non-airtight packages
- Gas pressurization from radiolytic and thermal degradation of inadequately stabilized materials and organic constituents

Four key considerations for safe storage of oxide and metal are identified:

- Adequacy of the calcination process
- Container resistance to pressure
- Container sealing requirements
- Container resistance to corrosion and radiation

The evaluation shows that rational explanations exist for all documented failures and that the associated conditions are well addressed by the requirements of DOE-STD-3013, for materials applicable to this standard. Since vulnerability studies were conducted in 1994 and appropriate

corrective actions were taken, only one significant DOE actinide storage container failure for plutonium oxide or metal is known, and that resulted from an inadequate closure weld in a singly-contained package.

1. Introduction

Storage of plutonium oxide and metal has been necessary since the inception of large-scale nuclear materials processing more than fifty years ago. However, it largely has been within the last twenty to thirty years that significant quantities of plutonium-bearing materials have been stored for extended periods at U.S. Department of Energy (DOE) facilities outside of nuclear weapons and components. The plutonium environment can be hostile with regard to package integrity. A number of package failures involving plutonium metal, oxide and residues have been well documented in a series of summary reports, reviews and popular articles. Examples are given in references 1-8. Factors that contributed to failures include container corrosion, gas pressurization, and volume expansion due to metal oxidation. Safety concerns about such vulnerabilities led to the issuance in 1994 of Recommendation 94-1 by the Defense Nuclear Facilities Safety Board (DNFSB).[9] In response, DOE prepared an implementation plan to address the vulnerabilities.[10] The implementation plan was revised in 1998.[11] Related recommendations on uranium-233 (DNFSB 97-1) and plutonium (DNFSB 2000-1) have been issued more recently.[12,13]

The five major plutonium sites in the DOE complex have been Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), the Hanford site, the Rocky Flats site (RFS) and the Savannah River Site (SRS). While some notable operationally significant package failures of plutonium oxide and metal have occurred at these sites, it is also noteworthy that tens of thousands of packages did not fail during decades of operation, despite the lack of standardized stabilization and packaging protocols in the past. Valuable lessons were learned in assessing the storage successes as well as the many fewer failures. Improved surveillance procedures also were implemented. In general, a reduction in failure frequency and consequences resulted complex-wide. Indeed, since vulnerability studies were conducted in 1994 and appropriate corrective actions were taken, only one significant DOE plutonium oxide or metal storage container failure is known. That failure resulted from an inadequate closure weld in a singly-contained package. The recently approved plutonium stabilization, packaging and storage standard DOE-STD-3013-2000 (referred to hereinafter as STD-3013) and its predecessors used these lessons learned to specify criteria for safe fifty-year storage.[14-17]

The purpose of this present article is to consolidate the well-documented reports of package failures and to place them in the context of overall storage success and requirements of STD-3013. This analysis is restricted to materials categorized as oxides (> 30 wt.% plutonium) and metal applicable to this standard. Not comprehensively addressed in this study are failures of unstabilized plutonium-bearing residues and wastes that are not directly pertinent to safe storage of oxide and metal or STD-3013. A number of failures of this type are cited in References 1-8. A recent example of the failure of a package of unstabilized residue is the August 5, 2003, event at the LANL plutonium facility. That event involved unstabilized cellulose residues bearing plutonium-238 in a slip-lid can/plastic bag storage configuration. Details are provided in a type B Accident Investigation report issued by DOE in December 2003

For the purpose of this report, container failure is defined as compromise of the package's main safety function, specifically containment of radioactive material. Tables 1 and 2 summarize all well-documented instances of plutonium oxide and metal storage package failures from our information search. In this report we also discuss some documented cases of unusual storage occurrences, such as collapsed ("paneled") or bulged rim-sealed food-pack cans, where an unusual condition was noted but contamination was not released. Examples of such cases are summarized in Table 3.

In the past, long-term storage of oxide and metal generally was not a practical concern due to the demand for plutonium. For package failures and unusual occurrences before about 1970, documentation is sketchy at best and very little written record exists. Undocumented failures undoubtedly occurred in this early period but are lost to history. In the present study, subject matter experts were surveyed in an attempt to capture early significant failure incidents that may not have been documented. Documentation has improved dramatically since the 1970's and has continued to improve to the present day. We believe it is unlikely that any major failure within the United States or United Kingdom since the mid 1970s would have been missed in the information search. The authors are keenly interested in being apprised of any relevant incidents that may have been overlooked.

In 1994, DOE adopted a consensus standard for packaging plutonium metal and oxide materials that contain greater than fifty weight percent plutonium.[17] The objective was to avoid container failures during a storage period of fifty years, with minimal surveillance. In 1996, 1999 and 2000, revisions were issued.[15-17] The latest revision is referred to as STD-3013 in this discussion. Among other changes from the earlier standards in this sequence, the current STD-3013 lowers the acceptable minimum actinide content from fifty to thirty weight percent, eliminates any constraint on maximum material temperature and reduces the maximum acceptable wattage per package from thirty to nineteen watts. Appendix A of STD-3013 outlines the technical basis for these changes. This report further supports the technical basis by evaluating documented plutonium storage incidents within the context of requirements of the standards.

Discussion of the dominant failure modes and safe storage considerations form the focus for the remainder of this report. A 1999 Los Alamos report provided a preliminary account of this work.[18]

2. Sources of Information

Valuable information for this report was obtained from direct discussions with many active subject matter experts at DOE's five principal plutonium-handling sites. Subject matter experts from other DOE sites, retired personnel, and knowledgeable managers from the United Kingdom's Atomic Weapons Establishment (AWE) also were engaged. Some of these subject matter experts are identified at the end of this report. Published information that was surveyed included DOE-wide plutonium storage safety evaluations, "grey-literature" technical reports and the peer-reviewed scientific literature. A literature search was conducted using the following keywords: plutonium, storage, package, failure, metal, oxide, compounds, and residues. The following databases were searched: INSPEC, Engineering Database, and DOE Energy Science and Technology Database.

A search of DOE's Occurrence Reporting and Processing System (ORPS) electronic database also was conducted. The ORPS database search produced no information not acquired through the other means mentioned above.

3. Container Failure Events and Mechanisms

The information search revealed documented plutonium storage package failures relevant to STD-3013, presented as case studies in Tables 1 and 2. Table 3 lists examples of documented unusual occurrences that did not result in release of contamination from the storage package. Photographs of some failed or off-normal containers are shown in Figures 1-5. Figure 6 shows a cutaway view of a STD-3013-type package. Two dominant observed failure modes were noted (see section 6 for further details) and are discussed in this section. A few examples of each failure mode are highlighted in the discussion.

a. Metal Oxidation in Non-airtight Packages. The largest number (nine) of well-documented package failures involved storage of plutonium metal in containers that were not air-tight (Table 1). In most cases, in-leakage of air led to oxidation of the metal to the dioxide, accompanied by a large increase in material volume that eventually caused mechanical failure of the container. Excellent descriptions of several events of this type are given in references 4 and 19-21. The roles of moisture, hydriding, nitriding and atmospheric pressure cycling in accelerating oxidation rates are elucidated in these reports.

To illustrate the metal oxidation failure mode, we cite an incident at LLNL which was discussed in detail by Dodson and summarized as Case SEAL-5 in Table 1.[20] In this instance, air entered an inner aluminum can through incomplete sealing of the container, followed by conversion of the plutonium metal to oxide and mechanical failure of the container. Failure occurred within three years of packaging. The can was found to be split along its entire length as a result of expansion of the oxidized metal. Figure 1 shows a photograph of a container that failed similarly at RFS in 1982 (Case Seal-4, Table 1).

A September 1999 event at SRS (Case SEAL-9, Table 1) provides a special case of metal oxidation in non-airtight packages. To the author's knowledge, this is the only example of a significant failure of an actinide metal or oxide storage package since vulnerability assessments were published in 1994. In this instance, a defective closure weld was made on a stainless steel container enclosing a metal button.[22,23] A photograph of the defective weld is shown in Figure 2. The weld inspection and testing process failed to detect this defect. The non-airtight container was placed into vault storage without secondary containment. Fifteen months later when the container was handled, extensive vault contamination and plutonium ingestion by seven workers occurred. Inspection of the container contents showed that extensive oxidation of the metal occurred during storage, generating easily-suspendable oxide in the container that moved through the weld defect when the container was handled.

b. Gas Pressurization. As indicated in numerous technical reports and publications, failures of packages containing plutonium oxide have occurred because of excessive gas generation. Examples are given in references 2-4. Table 2 lists the seven documented cases of this failure mode. The root causes stem from radiolytic and thermal degradation of inadequately stabilized material or the presence of organics including plastic. Figure 3 shows a photograph of a Hanford container that failed in 1980 by gas pressurization (Case Pressure-5, Table 2).

The gas pressurization failure mode also is illustrated by incidents at the Hanford Plutonium Finishing Plant (PFP) in 1975 and 1984 (Cases PRESSURE-1 and 2 in Table 2). These incidents involved unstabilized glovebox sweepings packaged in rim-sealed cans. In both cases, gas pressurization and rupture of the containers occurred. In one case the container was ejected from its storage position and gross contamination of the storage vault resulted. The other failure occurred inside a shipping container and resulted in gross contamination of the interior of the shipping container.

An example of failure due to organics degradation is the 1980 SRS incident listed as Case PRESSURE-6 in Table 2. In this case, an oxide storage package ruptured due to overpressurization, resulting in contamination of a large area of a storage vault. The stored material consisted of glovebox sweepings and reject pressed compacts containing plutonium dioxide in contact with an aluminum stearate-dodecanol die lubricant. Inspection of similar packages indicated pressurization from buildup of hydrogen and methane due to radiolytic and/or thermal degradation of the organic material.

4. Unusual Storage Occurrences Without Failure

Table 3 tabulates examples of documented cases in which unusual conditions were noted but storage package failures did not occur, i.e. no contamination was released. "Bulging" due to internal gas pressure and "paneling" due to partial vacuum of food-pack cans dominate this category of events. Figure 4 and 5 show photographs of paneled and bulged food-pack cans.

In a 1994 example from SRS (Case PRESSURE-11, Table 3), several food-pack storage cans were observed to be slightly deformed from small internal pressure buildup. The most probable cause of the pressurization was postulated to be a combination of thermal and radiolytic degradation of the PVC bag enclosing the inner container, with a possible small contribution from heating of the can atmosphere. At the United Kingdom's Aldermaston Weapons Establishment, pressurization of food-pack containers of plutonium oxide has only been observed for two containers in recent years, and these were packaged elsewhere under uncertain conditions.[29]

As Table 3 indicates, partial sidewall collapse ("paneling") or inward lid deflection of food-pack cans has been observed at PFP and SRS during storage of alpha-phase fuels-grade plutonium metal. An observation at SRS in 1998 (Case PANEL-6, Table 3) apparently involved scrap mixed oxide with high-burn up plutonium metal containing incompletely calcined carbide. Likewise, paneling has been observed at Aldermaston only with high-burn up plutonium metal, but not with oxide or weapons-grade metal.[29] A number of reports describe creation of vacuum from reaction of oxygen and nitrogen from air cover gas with plutonium metal (e.g., see References 4, 31, and 32 and references cited therein). None of the paneling cases listed in Table 3 led to release of contamination and no instances are known to the authors for weapons-grade metal or metal phases other than alpha. The experience strongly suggests the importance of elevated temperature and reactive metal (alpha phase) for the paneling process.

5. Generally Declining Failure Frequency

For the documented cases presented in Tables 1 and 2, a general decline in the frequency and consequence of package failures in recent years is evident. The 1999 SRS incident (Case SEAL-9, Table 1), due to a quality assurance failure, as discussed above, is a notable exception to this rule. Table 3 indicates a greater recent frequency of unusual occurrences observation without failure, but this is most likely a result of more aggressive surveillance and reporting in recent years. The general decline in failure rate is attributable to the development and application of improved stabilization and packaging protocols from applying lessons learned from previous packaging failures and successes, in combination with improved surveillance. Forums such as the

1984 DOE training seminar "Prevention of Significant Nuclear Events", the March 1999 American Chemical Society symposium on 50-year storage of nuclear materials and active complex-wide working groups have provided valuable mechanisms for information exchange in this regard.[33]

It is noteworthy that Dodson's 1994 report[20] indicated that only three package failures had been documented or remembered by facility personnel between the start of plutonium operations at LLNL in 1961 and the publication of her report. Only one of these failures was discovered during processing of more than 606 packages containing plutonium during an inventory reduction campaign. No failures have been observed at LLNL since completion of this campaign.[34] Several unusual occurrences without contamination release (e.g., bulging cans containing impure oxides that had not been processed according to the standards) have been reported by LLNL, as indicated in Table 3.

In the mid-1990s, a visual inspection of LANL's entire vault inventory of nearly 8000 plutonium items was conducted.[35] This exercise found that 361 containers had some visually observable abnormality. Of these, 82 containers had lost secondary (outer) containment as indicated by raised lids, corrosion or other factors. However, no containment losses occurred that dispersed material outside the packages. Indeed, during about 25 years of operation of LANL's plutonium facility, no containers of oxide or metal have failed in an uncontrolled environment.[36] The most commonly observed cause of package abnormalities has been mechanical, for example, a bagout bag pushing against a taped slip-lid. A few cases of primary container failure involved corrosive or inadequately dried materials. None of the containers that lost primary containment had been stabilized or packaged in a manner consistent with the requirements of STD-3013, and all of these cases have rational explanations well outside the requirements of the standard.

It is important to note, however, that the overall recent success in safely storing plutonium in vault environments have involved lower temperatures than for some postulated bounding storage scenarios after packaging according to the STD-3013 and did not universally involve welded closures.[37,38] Specifically, these hypothetical scenarios involve long-term storage in shipping containers in facilities that are not actively cooled. Some temporary off-normal scenarios assume extreme exposure of the shipping containers to direct solar radiation.

6. Critical Storage Standard Considerations

In this section, four key considerations for safe storage of plutonium oxide and metal are discussed within the context of STD-3013 and the two dominant observed failure modes discussed in Section III. The four key considerations are:

- Adequacy of the calcination process
- Container resistance to pressure
- Container sealing requirements
- Container resistance to corrosion and radiation

a. Adequacy of the Calcination Process. For oxide materials, STD-3013 requires calcination at 950°C for two hours to ensure elimination of gas-generating constituents such as organics, oxalates and nitrates as well as moisture. These requirements are intended to eliminate all significant sources of gas pressurization. Accordingly, failures and unusual occurrences of this type should be eliminated. The moisture content is required to be lower than 0.5 wt.% at the time of packaging to limit the potential for pressurization from hydrogen and/or oxygen generation.

As indicated in Appendix A of STD-3013, this water limitation is considered effective in this regard. However, it should be noted that the extent and consequences of water equilibria involving oxides that are likely to occur at some temperatures and water contents of interest have not been evaluated exhaustively and the possibility of condensed water inside the containers as a result of some bounding transportation scenarios has not been precluded categorically.[39,40] Research activities at LANL are actively evaluating such possibilities.

b. Container Resistance to Pressure. The plutonium storage container must survive or prevent four types of pressure scenarios:

- Gas vacuum
- Gas pressurization
- Material volume expansion due to metal oxidation
- Metal volume expansion due to phase changes

Tables 1-3 show failures and unusual conditions corresponding to the first three pressure scenarios.

The first pressure scenario (gas vacuum) is addressed in STD-3013 by specification of a storage container with sufficient mechanical strength to withstand total internal vacuum (0 psia).

The second pressure scenario (gas pressurization) is addressed in several ways in STD-3013. First, a package design working pressure of 699 psia is specified. The measured burst pressure of the package is nearly two orders of magnitude greater than the burst pressure of food-pack cans commonly used in the past.[41-43] In addition, to minimize the potential for gas generation, the standard requires calcination at 950°C to eliminate potentially problematic constituents. The standard also requires testing to ensure that water content of the packaged materials is below 0.5 wt.%. These conditions were not assured for any of the gas pressure-induced incidents listed in Tables 2 and 3.

The 1998 peer review report of the U.K. Aldermaston Weapons Establishment (AWE) interim storage criteria for plutonium-bearing materials contains sections written by representatives from most major DOE plutonium facilities.[44] The sections indicate that, in the experience of the reviewers, no containers of oxide produced and packaged in a well-controlled, reasonably dry atmosphere at a temperature of 400°C or above has exhibited significant container pressurization, even though a loss on ignition value below 0.5 wt. % may not have been attained at this relatively low stabilization temperature.

Standard 3013 addresses the third pressurization scenario (oxidation of metal) by requiring the use of nested, welded and leak-tested containers to essentially eliminate the possibility of exposing contained metal to air. The greater mechanical strength of the packages compared to food-pack cans also greatly enhances resistance to mechanical failure even if air in-leakage were to occur.

The fourth potential pressurization scenario (metal phase change effects) stems from a concern that volume expansions that occur when plutonium metal phase transformations occur near 115°C (alpha/beta) and 185°C (beta/gamma) may exert sufficient mechanical pressure to cause the storage container to fail. Our information survey revealed no documented or anecdotal evidence for this failure mode in containers with far less mechanical strength than those required by standard STD-3013. The metal phase change concern has been eliminated by worst-case experiments and finite element modeling.[45-47]

c. Container Sealing Requirements. As discussed in the preceding section, STD-3013 requires that mechanically strong, nested, welded and leak-tested stainless steel containers be used for packaging plutonium metal and oxides for extended storage. This requirement is intended to ensure that the containers will be adequately sealed to minimize or eliminate the possibility of air in-leakage. A robust quality assurance program for container sealing also is required by STD-3013.

d. Container Corrosion and Radiation Resistance. With one possible exception (Case PRESSURE-3, Table 2), none of the oxide and metal storage package failures and unusual occurrences summarized in Tables 1-3 have been caused by corrosion. (Numerous corrosion-related failures of unstabilized residue and waste packages have occurred.) STD-3013 minimizes the possibility of corrosion-related failures by allowing only stabilized oxides and metal to be packaged. In addition, the standard specifies that corrosive constituents be excluded and that corrosion-resistant container materials (e.g., L-series stainless steel) be used.

The issue of chloride-induced corrosion of storage containers has been addressed by Kolman.[48] STD-3013 and its predecessors do not specifically exclude chlorides. Kolman's key conclusion is that neither general corrosion nor stress corrosion cracking should pose a threat to 3013 containers under anticipated storage conditions, provided condensed water is avoided. The calcination, moisture and sealing specifications of the standard are intended to avoid any possibility of condensed water in the packages. However, as mentioned above, recent results indicate the need for additional evaluation of moisture equilibria at bounding temperatures and water contents of interest.[39,40]

Kolman also addresses radiation effects on the stainless steel container. His key conclusion in this regard is that radiation effects are unlikely to be a significant safety issue if good welding practices are followed.

A recent report on chloride salt radiolytic effects in plutonium storage environments surveys complex-wide experience in storing pyrochemical salts.[49] This survey indicates that significant corrosion problems have not been observed in storage of pyrochemical salts, provided reasonable precautions had been made to avoid excessive moisture. For example, Hanford has stored plutonium-bearing NaCl-KCl salts in rim-sealed containers for nearly twenty years without significant storage problems (corrosion or otherwise).[50] These observations are supported by recent observations on pyrochemical salts at RFS, LANL, LLNL and AWE.[29,36,50-52] Again, it should be noted that much or all of this favorable experience with storage of plutonium/salt mixtures has been at temperatures lower than some postulated bounding conditions for STD-3013 packages.[37,38]

7. Conclusions

The evaluation described in this report shows that rational explanations exist for all documented cases of failure of storage packages containing plutonium oxide and metal. All documented failures have involved conditions that are fully addressed by requirements of STD-3013. Two root causes of documented failures are identified. One cause is volume expansion from oxidation of stored metal in non-airtight packages. The second cause is gas pressurization due to radiolytic and/or thermal decomposition of inadequately stabilized materials. The overall decrease in failure frequency observed in recent years is attributable to improved packaging and surveillance protocols developed by applying valuable lessons learned from earlier packaging failures and successes. These lessons learned have been addressed fully in STD-3013.

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For several years, Los Alamos National Laboratory has led the research and development project to resolve technical issues in implementing STD-3010. A crucial element of this program is the MIS project, in which representative materials from the principal DNFSB 94-1 sites are subjected to detailed characterization. The MIS effort strives to anticipate undesirable conditions that might arise during stabilizing, packaging and storing plutonium materials at the respective sites.

An essential element in focusing the MIS effort and interpreting the resulting information is an active working group comprised of senior subject matter experts with significant management and technical responsibilities at the major DOE plutonium sites. Weekly teleconferences, quarterly multi-day workshops, frequent technical data and report reviews, and other frequent information exchanges form the core of this effort. The membership of the MIS Working Group as of March 2004 is as follows:

Hanford Plutonium Finishing Plant	Richard W. Szempruch and Theodore J. Venetz
Lawrence Livermore National Laboratory	Karen E. Dodson
Los Alamos National Laboratory	Richard E. Mason
Rocky Flats Environmental Technology Site	Jerry L. Stakebake
Savannah River Site	James W. McClard and Jeffrey B. Schaade

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TABLE 1. FAILURES FROM METAL OXIDATION OR CORROSION

Case Number (Reference)	Year/Facility	Case Details	Cause of Failure	Failure Avoided by STD-3013?
SEAL-1 Reference 5	1969 Hanford	A fuels-grade plutonium metal button weighing 2 kg oxidized and ruptured the food-pack can after 13 months in storage due to radial growth of oxide. Vault was grossly contaminated and personnel were contaminated upon entering the vault.	Leak in the sealed can allowed air in-leakage. The metal oxidized, resulting in radial pressure that caused container failure.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.
SEAL-2 Reference 5	1970 Hanford	A fuels-grade plutonium metal ingot weighing 2.2 kg oxidized and ruptured food pack can after about two years in storage vault. Can configuration was sealed can-plastic bag-taped slip lid can. Oxide formed in the can and the radial growth caused failure of can sidewall. Vault was grossly contaminated and personnel were contaminated and received internal uptake upon entry into vault.	Leak in the sealed can allowed air in-leakage. The metal oxidized, resulting in radial pressure that caused container failure.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.
SEAL-3 Reference 5	1972 Hanford	Plutonium metal oxidized and the food-pack can split open in glovebox. Powder accumulated outside can but contamination was confined to glovebox.	Leak in the sealed can allowed air in-leakage. The metal oxidized, resulting in radial pressure that caused container failure.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.
SEAL-4 Reference 19	1982 Rocky Flats	Two out of twenty seven 3-kg alpha plutonium cylinders breached their containers. Packaging was in aluminum cans with steel-crimp lids and stainless steel overpack. The overpack closure was a close tolerance fit lid sealed with silicone polymer sealant. These assemblies were submerged in water in experiments. The handling area was contaminated. Upon opening one of the ruptured containers for inspection in an air glovebox, the plutonium and corrosion products spontaneously ignited and the metal burned completely.	Leak in the sealed can allowed air in-leakage. The metal oxidized, resulting in radial pressure that caused container failure.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.

SEAL-5 Reference 20	1992 Livermore	A seamless aluminum can with screw type lid was filled with 1108 g of Pu metal in 1989. This can was bagged out of the glovebox and placed in a one-gal can for storage. After 32 months, the package was retrieved for processing and the contents of the gallon can transferred into a glovebox. Upon removal of the plastic bags, the aluminum can as found to have split lengthwise due to oxidation of the metal. Approximately 622 g of metal had oxidized.	Leak in the sealed can allowed air in-leakage. The metal oxidized, resulting in radial pressure that caused container failure.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.
SEAL-6 Reference 19	1992 AWE	A Pu metal button packaged in 1985 inside a screw-top aluminum can was bagged from a glovebox and placed in a metal food pack can with crimp sealed lid. By 1990, the Pu had gained only 3 g of oxygen but by 1992, the plutonium was totally oxidized. The increase in volume of the oxide exerted a radial pressure that destroyed the aluminum can and ultimately caused the food pack cans to rupture, contaminating the storage bin.	Leak in the sealed can allowed air in-leakage. The metal oxidized, resulting in radial pressure that caused container failure.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.
SEAL-7 Reference 53	1993 Los Alamos	In 1979, 2.5 kg of cast Pu metal was enclosed in a 2-in.-diam vessel made of steel tubing with welded end caps. The cylinder was bagged out of the glovebox and stored in an 8-in.-diam by 15-in.-tall steel can with taped slip-lid closure and stored in a vault. Upon movement of the item to a processing area 14 years later, the handler's protective clothing and a transfer cart became contaminated. The inner welded steel vessel had one end torn away. Evidence was not seen of plutonium metal; only yellow-green oxide powder was observed. Hydride-catalyzed Pu corrosion was suspected. Faulty weld on stainless steel container caused leak in the sealed can.	Faulty weld on stainless steel container allowed air in-leakage. The metal oxidized, resulting in radial pressure that caused container failure.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.

SEAL-8 Reference 19	1993 Los Alamos	In 1984, 5 kg of plutonium metal was removed from a glovebox in a plastic bag and placed in a second plastic bag inside a lead-lined can with a taped lid seal. Radiolysis of the plastic produced hydrogen, which reacted with the plutonium to form plutonium hydride and/or nitride. The container was opened inside a hood in 1993. Disruption of the brittle plastic caused a massive breach and spontaneous ignition occurred. Both the operator and hood were contaminated.	Comingling of incompatible materials (plastic and plutonium metal) led to formation of pyrophoric hydride. Inappropriate containment and handling led to release of the plutonium material.	Yes. The standards prohibit the presence of plastic materials in the storage package.
SEAL-9 References 22 and 23	1999 Savannah River Site	In 1998, a button of metal was placed in a stainless steel can, sealed with a weld and placed into storage without secondary containment. The weld was defective and the poor seal was not detected by the weld testing/inspection procedure. During storage, substantial metal oxidation occurred. When the can was handled more than a year after initial containment, extensive vault contamination occurred and plutonium uptake by seven workers resulted.	A defective closure weld was not detected and the container was stored without secondary containment. Air-inleakage led to formation of easily suspendible oxide, which was released through the defective weld when the container was subsequently handled.	Yes. Container sealing requirements (leak tested welded closure) and redundant barriers prevent air entry.

TABLE 2. FAILURES FROM GAS PRESSURIZATION

Case Number (Reference)	Year/Facility	Case Details	Cause of Failure	Failure Avoided by STD-3013?
PRESSURE-1 Reference 5,24	1975 Hanford	A can of less than 300 g of plutonium glovebox sweepings ruptured and ejected from storage position in vault. Container was a food-pack can sealed and placed in storage about four days before the event took place. Scrap powder from oxalate precipitation process oxide production line was involved. Some powder from the precipitator/ calciner glovebox, reported to be dry and free-flowing, was sealed out and stored without thermal stabilization. Visible oxide contamination of storage vault floor resulted.	Gas was generated from unstabilized oxide constituents. Material spilled from oxalate precipitation process in a glovebox was added to can without calcination.	Yes. The 500-600°C calcination temperature of the subject process produced tons of well-behaved PuO_2 that had measured LOI of <0.5%. The 950°C calcinations requirement of the standard far exceeds the demonstrated stabilization temperature for this type of material. Over a thousand containers of product with LOI (done at 450°C) in the range 0.2-1.0% were produced. These items have not presented pressurization problems in over 15 years of subsequent storage. The 950°C calcination requirement of the standard far exceeds the demonstrated stabilization temperature for this type of material.
PRESSURE-2 Reference 5	1984 Hanford	This case is very similar to case PRESSURE-1, involving food-pack can of plutonium glovebox sweepings. In this instance, the can was being stored in a shipping container that was closed but not bolted closed for shipment as only temporary storage was intended. The shipping container was so badly contaminated that it had to be discarded.	Gas was generated from unstabilized oxide constituents. Material spilled from oxalate precipitation process in a glovebox was added to can without calcination.	Yes. The 500-600°C calcination temperature of the subject process at Hanford produced tons of well-behaved PuO_2 that had measured LOI of <0.5% during the mid-1980s.
PRESSURE-3 Reference 5	1976 Hanford	A food-pack can of plutonium oxalate precipitate derived from analytical laboratory wastes was found to be slightly bulged and leaking an oily-appearing substance during storage vault. Slight contamination of the storage rack resulted.	Packaging of unstabilized material led to gas generation and corrosion of container. The food-pack containers were intended to hold dry, stabilized materials.	Yes. The 950°C of the standard is adequate to remove gas-generating constituents and corrosive materials are prohibited by the standards.

PRESSURE-4 Reference 55	1979 Hanford	A food-pack container of high decay heat plutonium oxide ruptured, releasing a plutonium oxide aerosol. The room, equipment, and three workers were contaminated. The can had been sealed and removed from the glovebox on the previous day and was in a shipping container for about 12 h just prior to the rupture. The material contained lumps up to 0.5-in. suspected of not being fully heated to the 450 C calciner temperature. The lumps may have been avoided during sampling, making the sample taken not representative. Analysis was specific for water and would not have indicated potential for nitrate decomposition.	Gas generation probably was caused by insufficient conversion of nitrate to oxide and was promoted by self-heating of the high-heat material.	Yes. The 950°C calcination temperature required by the standard is sufficient to decompose all nitrate constituents. Also, the container pressure rating prescribed by the standard is much higher than for food-pack cans.
PRESSURE-5 Reference 56	1980 Hanford	Enriched uranium/plutonium scrap oxycarbide material was contained in a 1-pound, slip-lid can and enclosed in two layers of plastic. The material had been stored for about 15 years in a brass vial thought to contain kerosene. As the item was taken from a glovebox after being packaged, the material spontaneously ignited causing the container to breach. The event took place within an hour of opening the brass vial. The materials spontaneously ignited, the can over-pressurized can and gross contamination of the room and personnel resulted.	Gas pressurization resulted from inadequately stabilized material, plus formation of pyrophoric products. Interaction with residual hydrocarbons was suspected.	Yes. The 950°C calcination temperature specified by the standard is sufficient to completely convert oxycarbides and organics to stable oxide products. Organics are forbidden in the STD-3013 package.
PRESSURE-6 Reference 25	1980 Savannah River Site	An oxide storage package ruptured due to overpressurization, resulting in contamination of a large area of the storage vault. The stored material consisted of glovebox sweepings and reject pressed compacts containing plutonium oxide in contact with an aluminum stearate-dodecanol die lubricant. Inspection of similar packages indicated pressurization from build-up of hydrogen and methane due to radiolytic and/or thermal degradation of the organic material.	Radiolytic and/or thermal degradation of organic material present in the plutonium oxide resulted in pressurization and rupture of the storage can.	Yes. The 950°C calcination temperature specified by the standard is sufficient to eliminate organic constituents and produce stable oxides.

PRESSURE-7 Reference 57	1979 Savannah River Site	During removal of cans from a welded stainless steel capsule a shipping container, pressure and contamination were released into and out of a plastic containment hut. Pressurization of the capsule occurred during post loading leak testing of the shipping container with helium. Porosity in the capsule closure weld allowed injection of helium into capsule. The capsule end broke away from the body during opening with a pipe cutter. Release of helium pressure from welded capsule during opening of capsule released plutonium oxide into the room and contaminated personnel.	The incident was caused by inadequate leak testing procedures and weld quality assurance.	Yes. Helium leak testing of both inner and outer containers at time of packaging provides assurance that helium cannot leak into container in subsequent testing. Quality assurance requirements on welds should ensure detection of inadequate welds.

TABLE 3. EXAMPLES OF UNUSUAL OCCURRENCES WITHOUT FAILURE

Case Number (Reference)	Year/Facility	Case Details	Cause of Failure	Occurrence Avoided by STD-3013?
PANEL-1 Reference 5	1975 Hanford	Several cans received in a single shipment of fuels-grade plutonium metal were found to be punctured, paneled, charred, or deformed inward. One such deformed can contained about 350 grams of corrosion product. Contamination was observed on the inside of several shipping containers.	High decay heat caused abnormally high temperatures in shipping container causing discoloration of cans. The high temperature also enhanced reaction of plutonium with air in the cans, causing paneling. Similar later occurrences indicated formation of plutonium nitride at similar temperatures.	Yes. The STD-3013 containers will be strong enough to withstand total vacuum, elevated temperature and anticipated mechanical impacts.
PANEL-2 Reference 58	1983 Hanford	Two cans containing fuels grade metal buttons were found to be paneled. The buttons had been in storage in food pack cans for 4 and 14 years at the time of the discovery. No contamination was released. Reference 58 also mentions two previous similar occurrences. One was Case PANEL-1 and the other was a single Hanford can found in 1981 to be paneled.	Partial vacuum from metal reaction with air was sufficient to cause the can to panel.	Yes. The STD-3013 containers will be strong enough to withstand total vacuum.
PANEL-3 Reference 20	1986 Livermore	Approximately 186 g of Pu scrap metal was bagged out of a glovebox in a pint-sized can, placed in a gallon can and stored in a vault. After 15 months in storage, the gallon can was found to have collapsed under vacuum.	Partial vacuum from metal reaction with air was sufficient to cause the can to panel.	Yes. The STD-3013 containers will be strong enough to withstand total vacuum.
PANEL-4 Reference 59	1995 Savannah River Site	Two vault stored items containing fuels-grade plutonium metal exhibited inward can wall paneling on the outer cans. No contamination was released.	Partial vacuum from metal reaction with air was sufficient to cause paneling.	Yes. The STD-3013ontainers will be strong enough to withstand total vacuum.
PANEL-5 Reference 60	1998 Hanford	One of the buttons discussed in Case PANEL-1 spontaneously ignited when the container was opened in an air glovebox in 1975. Oxidation was assumed to be complete when burning ceased. The resultant oxide then was placed in a food-pack assembly and stored. When examined thirteen years later, the outer can of one of the items was found to be paneled.	Partial vacuum from metal reaction with air was sufficient to panel food-pack can.	Yes. The STD-3013 containers will be strong enough to withstand total vacuum. The 950°C calcination temperature specified by the standard will be sufficient to convert all metal fines to stable oxide.

PANEL-6 Reference 30,61	1998 Savannah River Site	A can of scrap mixed oxide derived from Pu/U carbide from FFTF fabrication program was found to be paneled. The container had been stored for about 15 years and paneling had not been observed during previous routine surveillance. The material apparently contained residual Pu/U carbide.	Partial vacuum from carbide reaction with air was sufficient to panel the food-pack can.	Yes. The STD-3013 containers will be strong enough to withstand total vacuum. The 950°C calcination temperature specified by the standard will be sufficient to convert all carbide to stable oxide.
PANEL-7 Reference 60	1996 Hanford	Six paneled cans were observed in a population of 52 metal items examined by radiography. The cans contained metal ingots each with approximately 12 W decay heat. No container rupture was observed and no contamination was detected during handling to obtain radiographs.	Partial vacuum from metal reaction with air was sufficient to panel the food-pack can.	Yes. The STD-3013 containers will be strong enough to withstand total vacuum.
PANEL-8 Reference 30	1998 Savannah River Site	A food-pack container of oxide from LLNL was discovered to be paneled. The material was believed not to be fully oxidized and to contain small metal particles.	Partial vacuum from metal reaction with air was sufficient to panel the food-pack can.	Yes. The STD-3013 containers will be strong enough to withstand total vacuum. Also, the 950°C calcination temperature specified by the standard will be sufficient to convert all metal particles to stable oxide.
SEAL-9 Reference 62	1993 Savannah River Site	A can containing a Pu metal button was observed to steadily gain weight over a period of 4 years since original packaging. When opened, the inner can was found to have a defective seal. The oxide formed in the inner can filled the can but did not mechanically rupture the can. No contamination was released from the outer container.	A defective seal allowed entry of air into inner can with subsequent oxidation of metal.	Yes. The standard's container sealing requirements (leak tested welded closure) and redundant barriers will ensure that air in-leakage does not occur.
PRESSURE-8 Reference 63	1994 Livermore	During routine surveillance, two sealed packages, each consisting of concentric double food-pack cans containing calcined plutonium mixed oxide residues, were discovered to have bulging lids.	Gas was generated from inadequately stabilized materials.	Yes. The 950°C calcinations temperature specified by the standard is adequate to remove gas-generating constituents.
PRESSURE-9 Reference 64	1985 Hanford	A container of uncalcined mixed oxide gel sphere material was observed to be bulged.	Gas was generated from inadequately stabilized materials.	Yes. The 950°C calcination temperature specified by the standards is adequate to eliminate gas-generating constituents.

PRESSURE-10 Reference 66	1986 Hanford	A bulged can (most likely food-pack type) was observed. The nature of the contained material was not included in the event fact sheet.	Gas pressurization very likely resulted from inadequately stabilized materials.	Yes. The 950°C calcination temperature specified by the standard is adequate to eliminate gas-generating constituents.
PRESSURE-11 References 25-27	1994 Savannah River Site	Several food-pack containers were found to be slightly deformed, indicative of an internal pressure of about 10 psig.	Pressurization probably was caused by a combination of thermal and radiolytic degradation of PVC bags, with a possible small contribution from heating of the can atmosphere.	Yes. The standard does not allow organics in the storage package. Also, STD-3013 containers will be strong enough to withstand total vacuum.

Figure 1. Photograph of a rim-sealed aluminum container that ruptured due to excessive mechanical pressure from oxidized plutonium metal, due to a faulty seal. This event occurred at Rocky Flats in 1982 and is described as case Seal-4 in Table 1.

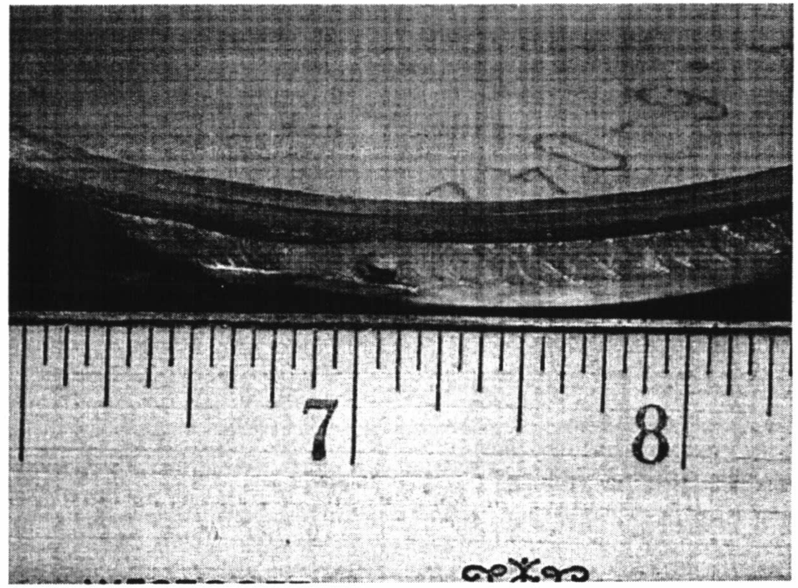

Figure 2. Photograph of a defective closure weld on a stainless steel storage container that led to an incident in September 1999 at Savannah River, causing uptake of plutonium by seven personnel and extensive vault contamination. This event is described as case Seal-9 in Table 1.

Figure 3. Photograph of a slip-lid can that ruptured due to excessive gas pressure. This event occurred at Hanford in 1980 and is described as case Pressure-5 in Table 2.

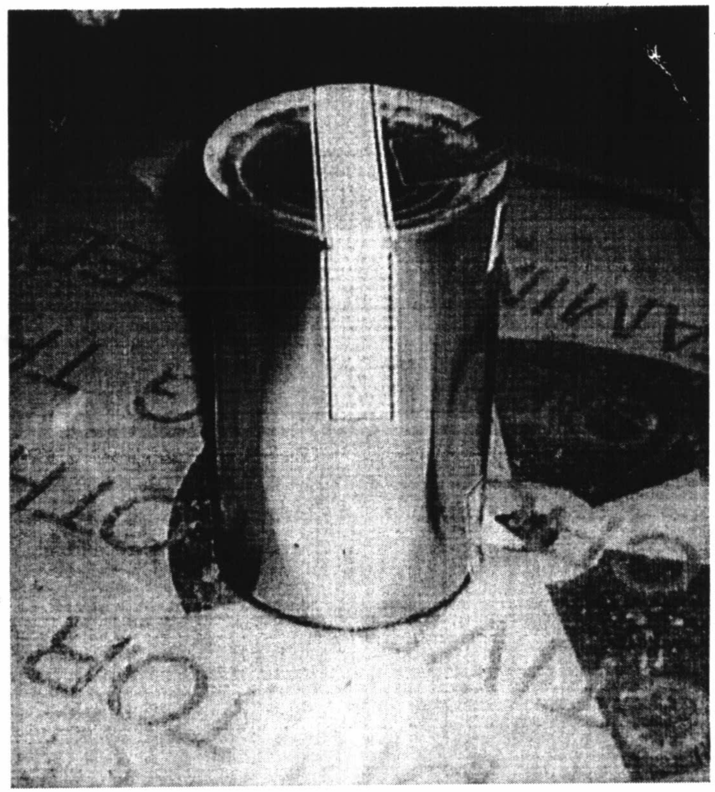

Figure 4. Photograph of a "paneled" rim-sealed Hanford container of plutonium metal that did not release contamination. Several examples of the paneling phenomenon are listed in Table 3.

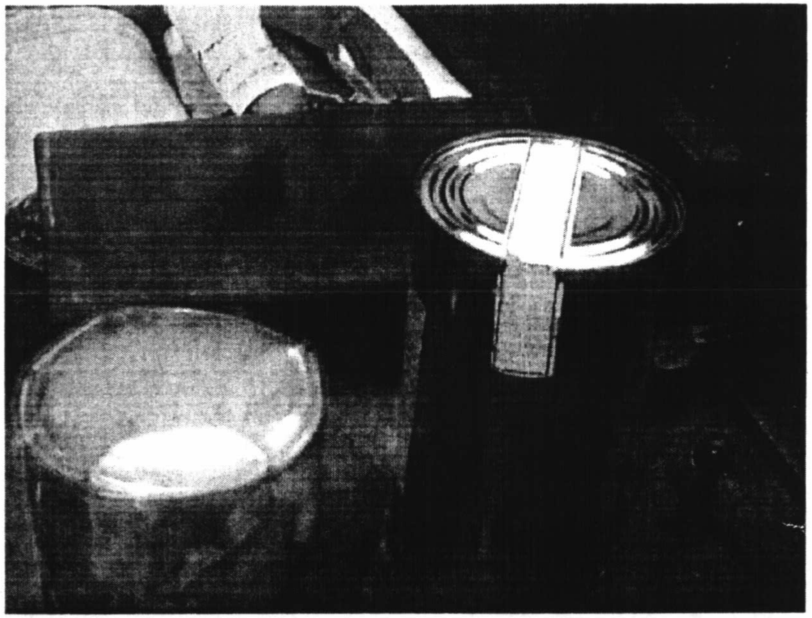

Figure 5. Photograph of a bulged Hanford rim-sealed container of plutonium oxide that did not release contamination. Other examples of the bulged can phenomenon are given in Table 3.

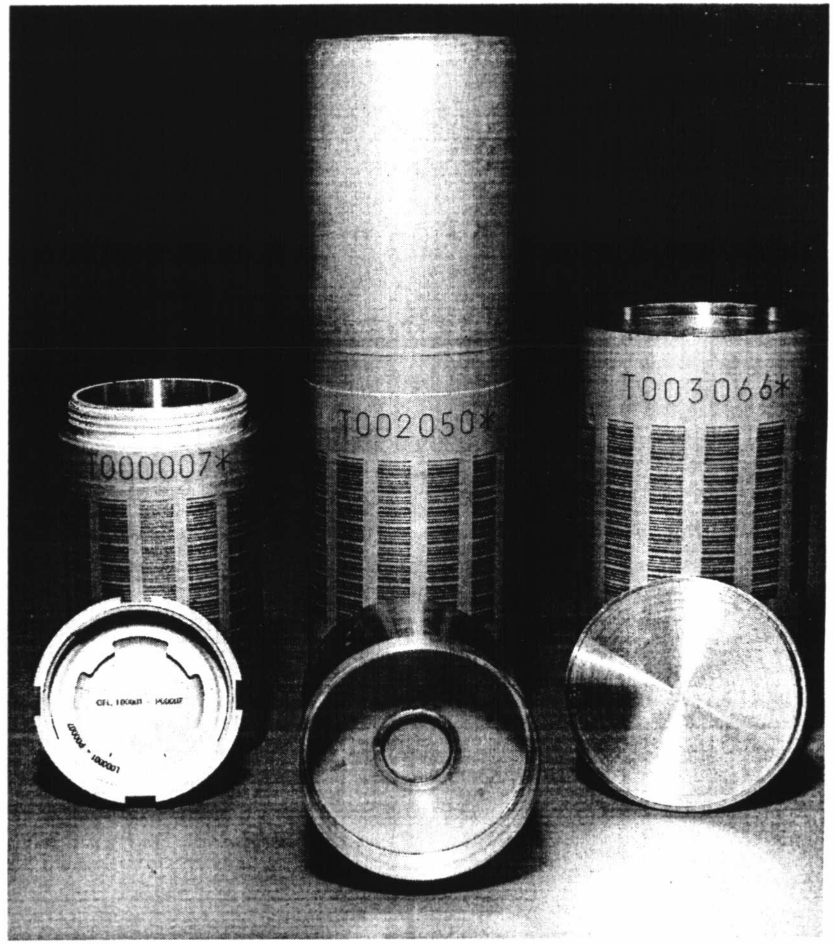

Figure 6. Photograph of a STD-3013-type package, showing a screw-cap inner convenience container and the weldable inner and .outer stainless steel containers.

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